

A Secondary Adhesionless Foundation with a Porous Structure Prepared using a Particle-Stabilized Emulsion

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This study addresses the common issue of foundation transferring onto clothes, particularly with liquid foundations (LF), and presents an innovative solution using particle-stabilized emulsion liquid foundation (PELF) to reduce secondary adhesion. Traditional approaches to mitigating transfer rely on enhancing evaporation, which can lead to undesirable effects such as skin dryness and powder aggregation. This research proposes an alternative approach inspired by the porous structure of diatomaceous earth: selectively drying the foundation surface to form a barrier against transfer while maintaining comfort and sensory appeal. PELF is made using hydrophobic silica particles to stabilize a water-in-oil emulsion, forming a porous structure as water evaporates. Unlike conventional surfactant-based liquid foundations (CSLFs) or oil-dispersed liquid foundations (ODLFs), PELF uniquely features particle-covered droplets that generate pores upon drying. Structural analyses using SEM and X-ray computed tomography confirmed the porous morphology of PELF, while CSLF and ODLF lacked similar porosity. Secondary adhesion resistance tests revealed that PELF outperforms CSLF under various drying times and pressures, showing a significant reduction in transfer by up to 90%. Higher water content in PELF formulations further enhanced adhesion resistance. Evaporation rate comparisons indicated similar initial rates for PELF and CSLF. Porosity, simulated based on aqueous phase ratios, correlated positively with adhesion resistance. The study concludes that the porous structure of PELF, enabled by particle stabilization, significantly improves secondary adhesion resistance without compromising texture. This technology holds potential for broader applications, including smudge-resistant sunscreens and makeup products, offering improved convenience and user satisfaction.

Key words: particle-stabilized emulsion, liquid foundation, secondary adhesion, makeup cosmetics, porous structure, 2-layer structure, drying time, volatilization, surfactants, X-ray computed tomography, SEM

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1. Introduction

When wearing white clothes, women often worry about foundation transferring onto collars. A 2018 questionnaire survey conducted in Italy, Japan, and China found that, on average, over 80% of women were uncomfortable with foundation staining their clothes (Fig. 1). Many reported that they intentionally restricted their daily activities to avoid such transfer and expressed a strong desire for foundation products designed to prevent secondary adhesion.

Liquid foundation (LF) is the most commonly used type of foundation worldwide. Secondary adhesion typically occurs either immediately after application or while getting dressed, due to the foundation not being fully dry and the friction between the foundation layer and clothing. Traditional methods to address secondary adhesion focus on speeding up the drying process of the foundation by increasing evaporation. However, these approaches often lead to undesirable consequences, such as the aggregation of powdery components and excessive skin dryness.

To prevent secondary adhesion due to friction, film-forming agents are often used to solidify the foundation layer.¹⁾ However, these solutions can leave an unpleasant texture on the skin, reducing their sensory appeal. This study proposes an alternative approach, allowing only the surface of the foundation layer to dry and solidify, rather than increasing evaporation rates.

The inspiration for this study came from diatomaceous earth, which is highly valued for its porous structure²⁾ and its ability to maintain dryness, as seen in applications such as absorbing water from wet feet on diatomaceous earth slates. Based on this property, we hypothesized that by incorporating a porous structure in the substrate, it would be possible to selectively dry the outermost surface. This dry outer layer could act as a barrier, reducing direct contact between the wet substrate and clothing, effectively preventing secondary adhesion.

In this study, the particle-stabilized emulsion (PE) technique^{3–5)} was used to achieve a porous structure throughout the foundation layer. Instead of using surfactants, solid particles were used to stabilize the oil-water interface. This method led to the development of a particle-stabilized emulsion liquid foundation (PELF) with numerous pores. The study subsequently investigated the relationship between the porous structure of PELF and its ability to prevent secondary adhesion.

2. Materials and Methods

2.1. Preparation of test samples

PE technology used hydrophobic silica particles to prepare a water-in-oil (W/O)-type PELF with a 50% internal water phase. The pigment and surfactant were first highly dispersed into the oil phase using a homogenizer (PRIMIX, Hyogo, Japan; Labolution) at 12000 rpm. Then, the water phase was slowly added over approximately 4 min while maintaining the same speed of 12000 rpm. Preparation was carried out at an 800 g scale using a 1-L beaker. The hydrophobic silica used was fumed silica with a primary particle size of 12 nm and a specific surface area of 200 m²/g. The surface was treated with a silylating agent, making it moderately hydrophobic. For comparison, a W/O conventional surfactant liquid foundation (CSLF) containing the same 50% internal water phase was also prepared (Table 1). Additionally, an oil dispersion liquid foundation (ODLF), containing oil and particulate materials without emulsification, was prepared to evaluate the effect of emulsifiers on secondary adhesion (Table 1). To examine the significance of the porous structure formed after particle adsorption and layer drying, the hydrophobic silica particles used in PELF were incorporated into CSLF and ODLF formulations. The presence and behavior of hydrophobic silica particles in these LF samples were analyzed by freeze-fracture scanning electron microscopy (SEM) with a HELIOS Nanolab 650 (Thermo Fisher Scientific, Portland, OR, USA).

2.2. Confirmation of the internal structure of the foundation layer

The surface structure of the foundation layer was evaluated by applying 2.0 mg/cm² of LF samples onto 50 mm square sheets of artificial leather (Saprare, Idemitsu Technofine, Tokyo, Japan) and allowing them to dry for 30 min at room temperature. The dried surfaces were then examined using a SEM (JSM-IT500HR In Touch Scope, JEOL Ltd, Tokyo, Japan). Additionally, the cross-section of the dried PELF layer was observed by an X-ray computed tomography (TDM1600H-Sμ/DD/F, Yamato Scientific, Tokyo, Japan).

2.3. Confirmation of the secondary adhesionless function

To assess the secondary adhesion-resistant properties of LF samples dried for different time intervals, 0.05 g of each sample was spread on a 50-mm square piece of artificial leather. The samples were left to dry for various durations. A 70-mm square piece of 100% pure white cotton cloth was pressed onto the dried sample. While maintaining consistent

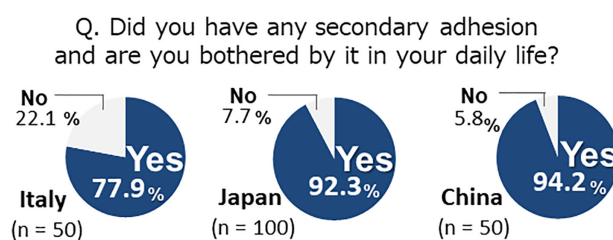


Fig. 1 Results of an online survey on secondary adhesion of foundation. A survey was conducted on 200 women who use foundation on a daily basis.

Table 1 Formulations of foundation used in this study (wt%).

Ingredient*		CSLF	ODLF	PELF
Oil phase	Trisiloxane(volatile)	20.0	70.0	20.0
	Dimethicone	9.2	11.6	11.6
	Cetyl ethylhexanoate	5.0	5.0	5.0
	Diphenylsiloxy phenyl trimethicone	5.0	5.0	5.0
Water phase	Water (volatile)	45.0		45.0
	Butylene glycol	3.0		3.0
	Glycerin	2.0		2.0
Surfactant	Silica silylate	1.2	1.2	1.2
	PEG-10 dimethicone	2.4		
Pigment	Titanium dioxide	5.0	5.0	5.0
	Iron oxides	2.0	2.0	2.0
	Triethoxycaprylsilane	0.1	0.1	0.1
	Aluminum hydroxide	0.1	0.1	0.1
Total		100.0	100.0	100.0

*All ingredients were commercially available cosmetic grade materials

CSLF, conventional surfactant-based liquid foundations; ODLF, oil-dispersed liquid foundations; PELF, particle-stabilized emulsion liquid foundation

pressure, the cloth was carefully pulled away. The areas of LF adhered to the cloth were photographed and analyzed using Photoshop CS6 (Adobe, San Jose, CA, USA). The adhered-to-total area ratio was calculated, with each formulation measured 3 times ($n = 3$). Data were expressed as the mean $\pm$ standard deviation.

To investigate the effect of “non-dryness,” the samples were tested after drying for 1, 3, 5, 10, and 15 min on the leather. At this time, the pressure was fixed at 9 kPa. The influence of applied “friction” between the sample layer and clothing was tested by pressing the white cloth on the artificial leather at pressures of 0.3, 0.6, 0.9, 1.2, and 1.5 kPa. The drying time was fixed at 5 min. PELF samples with internal water phase contents of 10%, 20%, 30%, 40%, and 50% were prepared to investigate the optimal porous structure volume. In addition, the fluctuation of water was replaced with trisiloxane. The experiment was carried out with a fixed pressure of 9 kPa and varying drying times. Their secondary adhesion-resistant properties were then evaluated. Notably, a 50% internal water phase was the maximum level for stable W/O emulsions. When the aqueous phase volume exceeded 50%, the sample separated and could not be prepared.

2.4. Measurements of evaporation rates of volatile components in CSLF, ODLF, and PELF

To measure the evaporation rates of CSLF, ODLF, and PELF, a 0.05-g sample was evenly spread on a 50-mm square sheet of artificial leather and left to dry for 30 min at room temperature. The weight of the sample spread on the leather was measured every minute over a 30-min drying period. The evaporation rates of volatile components were calculated using Equation (1)

$$\text{Evaporation rate (\%)} = (\text{Wt-int} - \text{Wt-spc}) / \text{Wt-int} \times 100 \quad (1)$$

where Wt-int and Wt-spc indicate the sample weights at the initial and specific times, respectively.

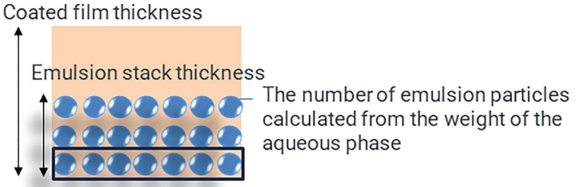
$$\text{Porosity (\%)} = \frac{\text{Emulsion stack thickness (\mu m)}}{\text{Coated film thickness (\mu m)}} \times 100$$


Fig. 2 Calculation formula and schematic diagram for porosity.

2.5. The simulation of porosity

Porosity is defined as the proportion of the emulsion structure in the thickness of the applied film. The calculation is done as follows (Fig. 2): 1) First, calculate the weight of one 5- μm diameter spherical emulsion droplet, and then calculate the number of emulsion droplets in the system from the weight of the water phase. 2) Calculate how many emulsion particles are spread in 1 cm^2 . 3) Then calculate how many layers are stacked to calculate the height of the structure in the film. 4) Calculate the thickness (height) of the sample applied per unit area. 5) Calculate the porosity by dividing the height of the structure by the thickness of the applied sample. Note that this calculation is based on the assumption that the fine particles used for emulsification are ideally dispersed and monodispersed, so they are much smaller than the droplet size of the emulsion particles and can be ignored. The diameter of the emulsion particles, 5 μm , is based on the approximate average value of the PELF samples used in the experiment.

3. Results

3.1. Preparation of LF samples and their internal structures

In the PELF and CSLF freeze-fracture samples, emulsified droplets with diameters ranging from 5 to 10 μm were observed. Notably, only the droplets emulsified with silica particles were surrounded by these particles (Fig. 3). Conversely, no particles were detected at the O/W interface in the CSLF sample, even when the same silica particles used in PELF were incorporated.

3.2. Observation of the surface structure of the foundation layer

Surface and cross-sectional analyses revealed that the PELF layer contained numerous empty pores, approximately 5 μm in diameter, likely formed owing to the evaporation of emulsified droplets (Figs. 4C and 5). Conversely, LF samples prepared using surfactant molecules or dispersion-based methods showed no such empty pores, while these samples exhibited aggregated particles, as shown in Figs. 4A and 4B.

Comparing Figs. 5B and 5C, there were many pores in the upper part and none in the lower part of the membrane that had not yet dried. In image 5C, pores with weak contrast can be seen, but these are emulsified particles and pores filled with water before becoming pores. This is because water has a lower X-ray transmittance than air, so it appears white. Furthermore, there are more hollow pores at the top, which indicates that this structure is formed by volatilization.

3.3. Confirmation of the secondary adhesionless function

As illustrated in Fig. 6, PELF demonstrated superior secondary adhesion resistance compared to CSLF over the entire drying period. This was evident on artificial leather. Especially during a 1-min drying period experiment simulating a “non-dry” scenario in daily life, PELF effectively reduced secondary adhesion far more than CSLF. In the white cloth pressing experiment, it was confirmed that the amount of secondary adhesion in CSLF and ODLF samples varied depending on the magnitude of the physical pressure. Figure 7 shows a clear correlation, indicating that higher physical pressure leads to increased secondary adhesion and a greater amount of foundation remaining on the cloth. On the other hand, PELF exhibited minimal sensitivity to pressure, maintaining consistently lower secondary adhesion levels than CSLF and ODLF even under higher pressure conditions. Figure 8 highlights the importance of the internal water phase in secondary adhesion resistance. Systems with higher water content displayed better adhesion resistance, emphasizing the vital role of water phase inclusion in achieving optimal performance. Compared to conventional LF, PELF succeeded in reducing the amount of secondary adhesion to 1/10.

3.4. Results of evaporation rates of volatile components in CSLF, ODLF, and PELF

The evaporation rate of ODLF was the lowest among the tested samples. As illustrated in Fig. 9, during the initial 0–1 min, there was no discernible difference in the evaporation rates of CSLF and PELF. After this period, the evaporation

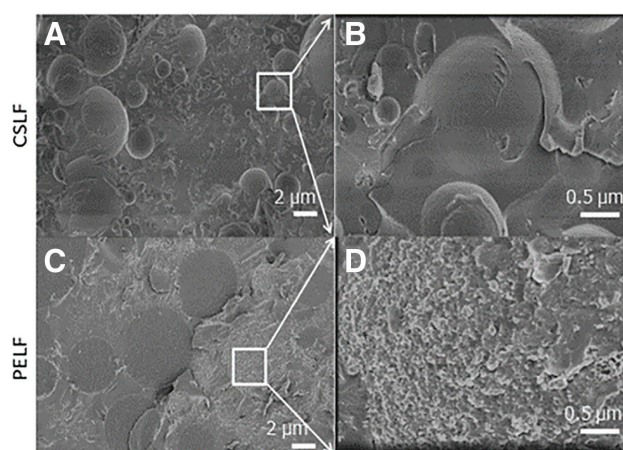


Fig. 3 Observation of particle-adsorption status in foundations by freeze-fracture SEM. Physicochemical statuses of emulsion droplets emulsified by silica particles or surfactant molecules, as well as the areas near their interfaces, were observed. Microphotographs A and B show the relatively wide area of the freeze-fracture surface and a closer look at the droplet interface in CSLF, respectively. Microphotographs C and D show the relatively wide area of the freeze-fracture surface and a closer look near the droplet interface in PELF, respectively. The bars in the wide area (A and C) and the bars in the magnified area (B and D) indicate 2 and 0.5 μm , respectively. CSLF, conventional surfactant-based liquid foundations; PELF, particle-stabilized emulsion liquid foundation; SEM, scanning electron microscopy

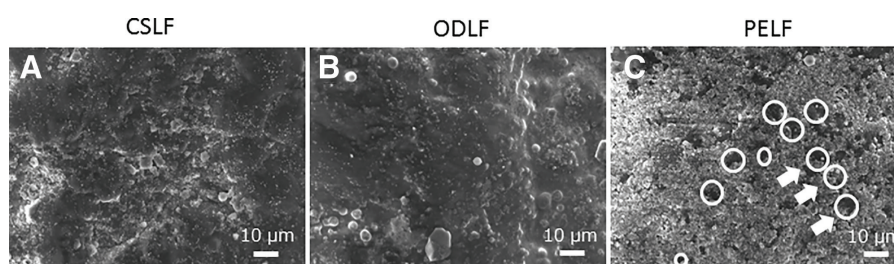


Fig. 4 Observations of the surfaces of foundation samples using SEM. Microphotographs A, B, and C show the surfaces of CSLF, ODLF, and PELF, respectively. The bars indicate 10 μm . In the PELF photograph, the white circles and arrows indicate empty pores formed after the evaporation of emulsified droplets. CSLF, conventional surfactant-based liquid foundations; ODLF, oil-dispersed liquid foundations; PELF, particle-stabilized emulsion liquid foundation; SEM, scanning electron microscopy

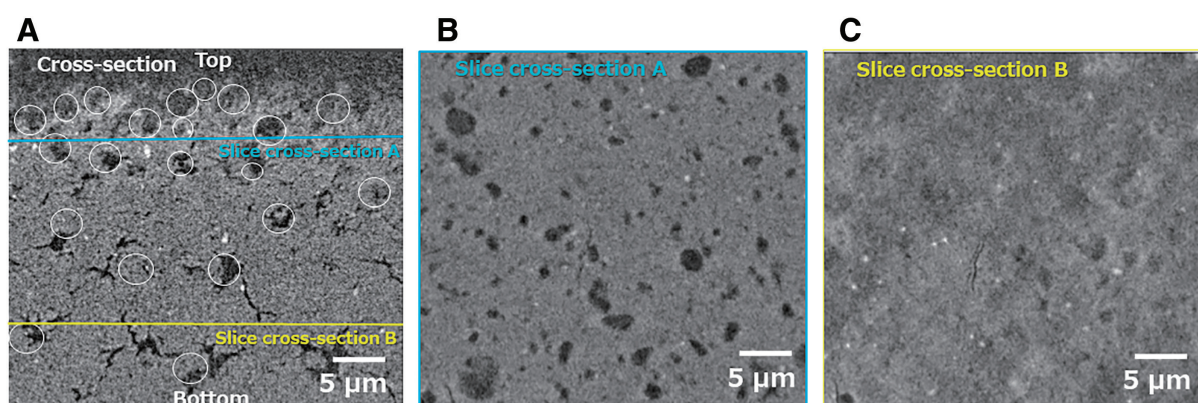


Fig. 5 Observation of the cross-section of the PELF layer by X-ray computed tomography. PELF was spread on artificial leather and dried at room temperature. The bars indicate 5 μm . The white circles indicate empty pores formed after the evaporation of emulsified droplets. (A) Cross-sectional view of the membrane, (B) slice cross-section of the upper part of the membrane, and (C) slice cross-section of the lower part of the membrane. PELF, particle-stabilized emulsion liquid foundation

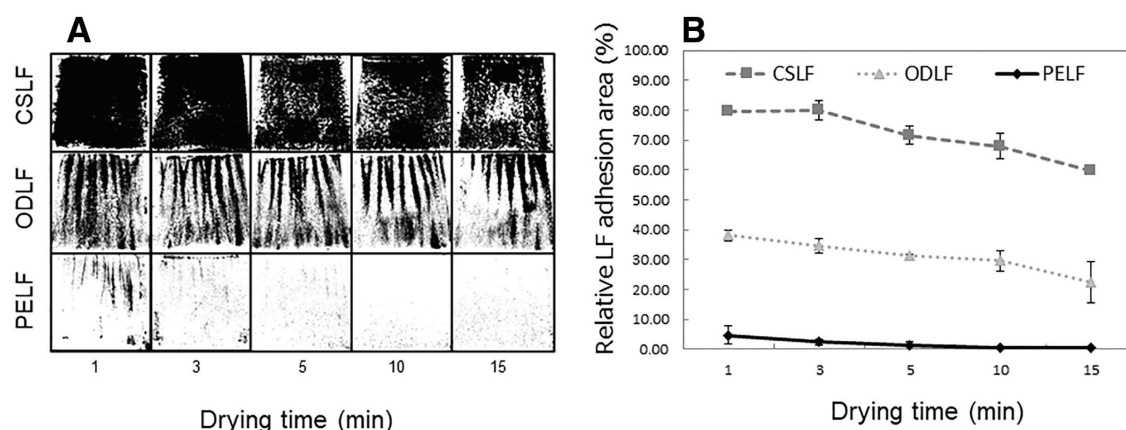


Fig. 6 Changes in the secondary adhesion resistance functions of LF samples based on different drying periods, illustrated by the ratio of secondary adhesion. (A) Macrophotographs present black-and-white images of LF residue adhered to the white cotton cloth. In graph (B), the y-axis shows the relative LF adhesion area (%), and the closed squares (■) with a dashed line, triangles (▲) with a dotted line, and the diamonds (◆) with a solid line representing the relative adhesion areas of CSLF, ODLF, and PELF, respectively.

CSLF, conventional surfactant-based liquid foundation; LF, liquid foundation; ODLF, oil-dispersed liquid foundation; PELF, particle-stabilized emulsion liquid foundation

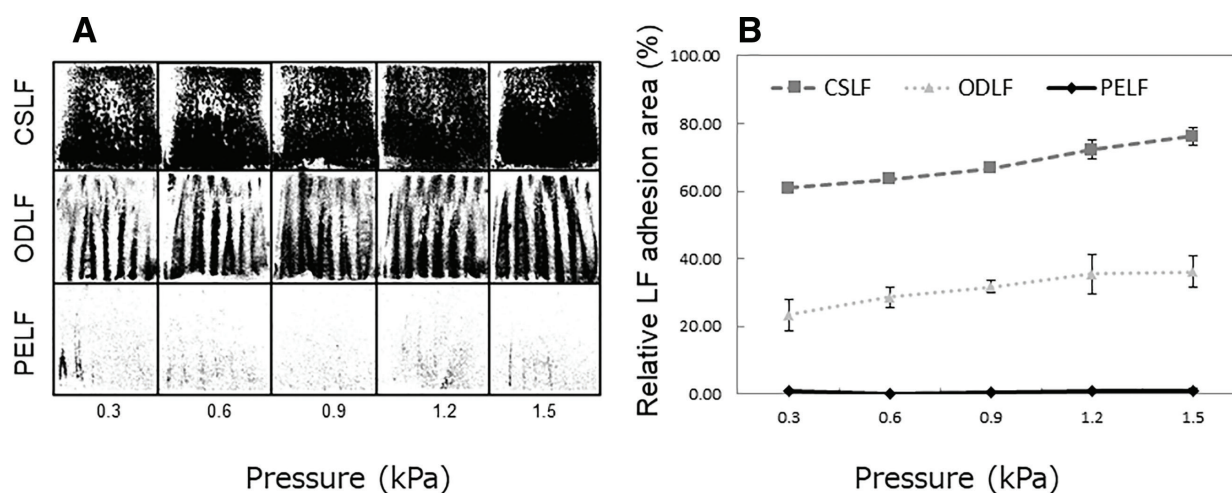


Fig. 7 Effect of pressure on the ratio of the secondary adhesion of LF samples. (A) Macrophotographs display black-and-white images of adhered LF samples on sheets of white cloth. In graph B, the y-axis shows the relative LF adhesion area (%), and the closed squares (■) with a dashed line, triangles (▲) with a dotted line, and the diamonds (◆) with a solid line showing the relative adhesion areas of CSLF, ODLF, and PELF, respectively.

CSLF, conventional surfactant-based liquid foundation; LF, liquid foundation; ODLF, oil-dispersed liquid foundation; PELF, particle-stabilized emulsion liquid foundation

rate of PELF decreased slightly, while the evaporation rate of CSLF increased slightly. However, there was little significant difference between them.

3.5. Simulated porosity in PELF samples

The results calculated from the internal aqueous phase ratio are as follows. The porosity of the PELF samples with aqueous phase ratios of 10%, 20%, 30%, 40%, and 50% was recorded as 31.4%, 44.8%, 58.2%, 71.6%, and 85.2%, respectively.

As shown in Fig. 10, it can be inferred that the more the aqueous phase, the higher the proportion of the emulsion structure within the thickness of the film.

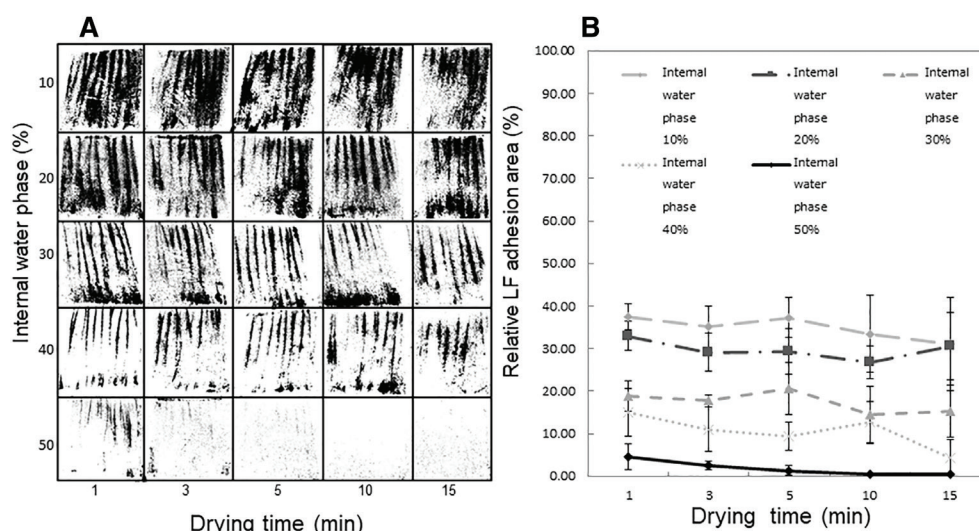


Fig. 8 Effect of the ratio of the internal water phase in PELF on the relative secondary adhesion. (A) Macrophotographs show the black-and-white images of adhered LF samples on the sheets of white cloth. In graph (B), the y-axis shows the relative LF adhesion area (%), and the closed diamonds (◆) with a dashed line representing an internal water phase 10%; the squares (■) with a dotted and dashed line represent an internal water phase of 20%; the triangles (▲) with a dashed line represent an internal water phase of 30%; the x-marks (x) with a dotted line represent an internal water phase of 40%; the closed diamonds (◆) with a solid line representing an internal water phase of 50%. LF, liquid foundation; PELF, particle-stabilized emulsion liquid foundation

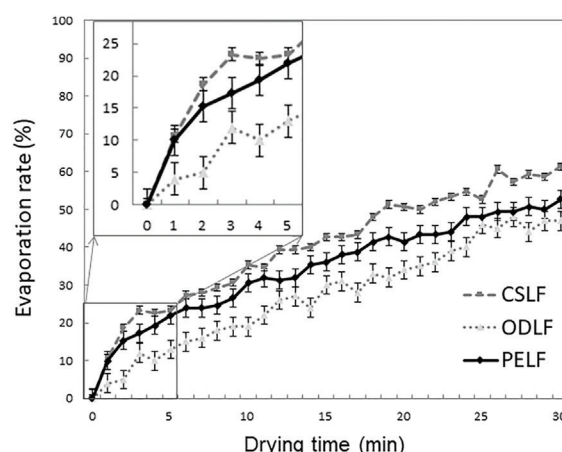


Fig. 9 Time courses of the evaporation rates of volatile components in CSLF, ODLF, and PELF. In the graph, the y-axis shows the evaporation rate, and the closed squares (■) with a dashed line, triangles (▲) with a dotted line, and the diamonds (◆) with a solid line showing the evaporation rates of CSLF, ODLF, and PELF, respectively. CSLF, conventional surfactant-based liquid foundations; LF, liquid foundation; ODLF, oil-dispersed liquid foundations; PELF, particle-stabilized emulsion liquid foundation

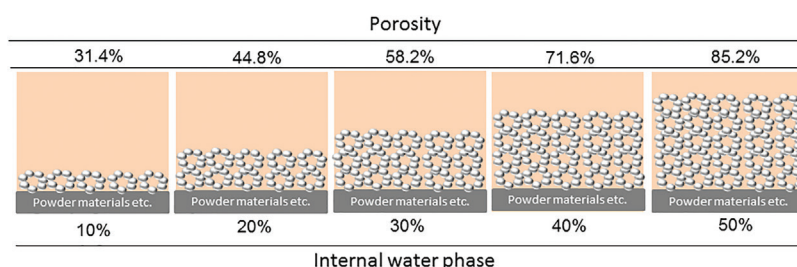
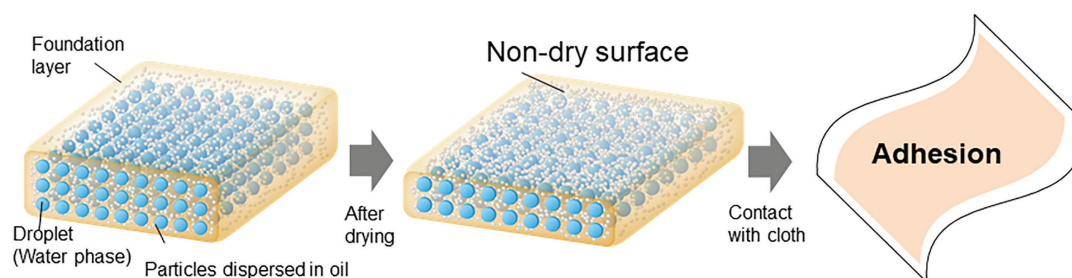


Fig. 10 Porosity simulations. The porosity of a specific foundation layer thickness, which was made by accumulating 5-μm water droplets in a W/O emulsion, was calculated. W/O, water in oil

Conventional surfactant liquid foundation (CSLF)



Particle-stabilized emulsion liquid foundation (PELF)

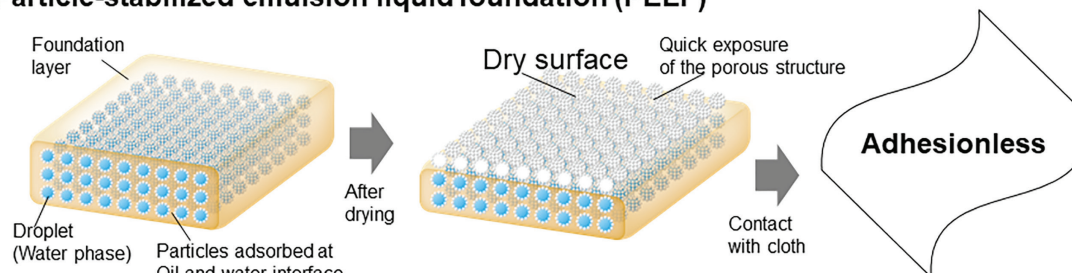


Fig. 11 Difference in the drying mechanisms of CSLF and PELF.

CSLF, conventional surfactant-based liquid foundations; PELF, particle-stabilized emulsion liquid foundation

4. Discussion

Products emulsified using the PE technique demonstrate remarkable characteristics compared to those made through conventional methods with emulsifiers.⁶⁾ In PE, the high adsorption energy at the interface ensures that once particles adsorb, they are challenging to desorb. The particles having small diameters used in PE tend to aggregate, leaving only the particles adhered to the interface as water and oil evaporate.⁷⁾ In PELF, the evaporation of the internal water phase creates numerous empty spaces surrounded by aggregated particles, resulting in a porous structure in the foundation layer.

The reduced evaporation rate of PELF after the first 2–3 min is attributed to the formation of an aggregate structure on the outermost surface as volatile components evaporate. After 1–2 min, when 10%–20% of the volatile components had disappeared, the solid structure made of silica particles began to become partially exposed in the sample with an internal phase ratio of 50% and an internal structure of about 85%. This aligns with experimental observations, which confirm that the secondary adhesion resistance function begins within a minute. By controlling PELF's evaporation time, the onset of the secondary adhesion resistance function can be adjusted as needed.

This study revealed 2 key findings: (1) Foundation samples with higher porosity, resulting from increased internal water content, demonstrated superior secondary adhesion resistance performance. (2) Despite having similar evaporation rates, CSLF and PELF differed significantly in their secondary adhesion resistance properties. These results suggest that the secondary adhesion resistance function arises from the multi-porous structure of PELF, which prevents direct contact between the foundation layer and cloth surfaces (Fig. 11). For conventional LF, liquid components in a non-dry state are absorbed by cloth upon contact, leading to secondary adhesion. Conversely, PELF's outermost layer prevents this, allowing it to demonstrate superior secondary adhesion resistance properties.

The secondary adhesion resistance function of PELF was found to remain unaffected by the intensity of “friction” between the LF layer and clothing. This highlights PELF's enhanced robustness and superior secondary adhesion resistance performance compared to conventional LFs. This is attributed to the fine silica particles, which aggregate firmly upon drying, thereby enhancing the structural strength of the entire foundation layer. When a liquid with high surface tension, such as water, evaporates, the cohesive force of the particles increases due to liquid bridging forces. Consequently, samples with a higher internal water phase demonstrated superior secondary adhesion resistance capability.

This study revealed that PE technology enables PELF to achieve the important property of suppressing secondary adhesion through “surface drying.” By leveraging these factors, the study achieved a remarkable reduction in secondary adhesion, decreasing it to just 1/10 of that observed with conventional LFs. These findings strongly support the

hypothesis that “surface drying” is the key to controlling secondary adhesion, and I believe that the functionality of diatomaceous earth, which inspired our original approach, can be successfully incorporated into our foundation.

5. Conclusion

This study employed a PE technique for preparing liquid foundations, replacing conventional surfactant-based methods. The resulting PELF layer exhibited a high secondary adhesion resistance function, even in non-dry and frictional conditions where conventional LFs often fail.

By adopting PELF technology, individuals can experience a more stress-free lifestyle, reducing concerns about secondary adhesion and enabling them to enjoy makeup and fashion with greater ease. Moreover, this technology has potential applications beyond foundations. For example, it could improve sunscreen formulations, making them more resistant to removal during physical activities or towel use in summer. Similarly, it could improve makeup products designed to resist smudging from children’s hands. Sharing the outcomes of this study on a global scale could enhance users’ comfort and happiness, offering a richer and more fulfilling daily experience.

Conflict of Interest: AN and KN are employees of POLA CHEMICAL INDUSTRIES, INC., which funded this study.

Abbreviations: CSLF, conventional surfactant-based liquid foundations; LF, liquid foundation; ODLF, oil-dispersed liquid foundations; PE, particle-stabilized emulsion PELF, particle-stabilized emulsion liquid foundation; SEM scanning electron microscopy

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